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Application of power ultrasound to cementitious materials: Advances, issues and perspectives

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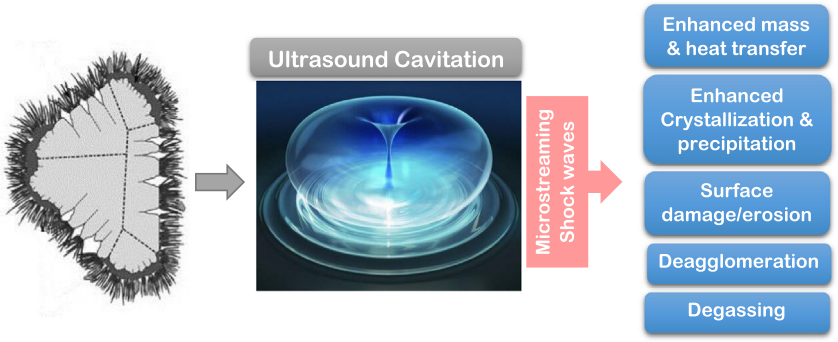
HIGHLIGHTS

- Effect of acoustic cavitation on sonocrystallisation and its application in cementitious materials have been discussed.
- It has been hypothesized that power ultrasound has a potential to enhance cement hydration due to the proposed mechanisms.
- Scarce current studies show that the PUS can improve the mechanical and microstructural properties of cementitious composites.
- Potential research needed to obtain a better understanding of the PUS interaction in cementitious materials have been suggested.

GRAPHICAL ABSTRACT



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ABSTRACT

Novel techniques such as power ultrasound (PUS) are currently under consideration to improve the hydration of cementitious materials and to promote the effectiveness of replacing supplementary cementitious materials; SCMs, in terms of mechanical, microstructural and transport properties. This could enhance the properties of cementitious composites, reduce the quantity of waste materials, as well as decreasing the CO_2 footprint of cementitious materials. A handful of studies have investigated this promising field and little is known about the mechanisms by which the ultrasound acts in cement-based systems. This paper outlines the possible mechanisms involved on the effects of PUS as a method to promote cement hydration kinetics of Portland cement and binary blends. It also reviews and analyses previous research conducted mostly on the dispersing effects of PUS on the enhancement of pozzolanic reactivity of SCMs in cementitious systems. This review concludes with some perspectives on research needed to gain a fundamental understanding of this emerging field.

1. Introduction

Traditional Portland cement-based concrete has provided the foundation for the built environment for almost 200 years. The focus of modern concrete structures is high performance, design for long lifetime and to be aesthetically pleasing. According to the Mineral Products Association (MPA) cement fact sheet [1], however, "the demands for

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sustainable development have placed a huge responsibility on the construction sector to continually improve existing processes, products and practices, and to innovate in order to reduce both energy used in service and embodied energy in products together with emission of greenhouse gases during manufacture".

The kinetic mechanisms of ordinary Portland cement (OPC) hydration and the complex, interdependent chemical and microstructural phenomena which control the characterisation of cement hydration products have not been completely understood. However, major efforts have been placed on the sustainable use and enhancing the perceived performance of OPC in cement-based composites.

Replacing part of Portland cement clinker by using alternative supplementary cementitious materials (SCMs) like fly ash (PFA), ground granulated blast furnace slag (GGBS), silica fume (SF), calcined clays and natural pozzolans in blended cements or utilising SCMs separately as a partially substitute additive in mortar/concrete systems represents a viable solution to reduce carbon footprint [2]. A few benefits have been investigated in cement-based materials incorporating SCMs including the improvement in consistency and the fresh concrete properties [3], decreased hydration heat evolution [4], improved mechanical/structural properties such as long-term strength development [5], enhanced durability [6–8] and reduced autogenous shrinkage [9]. However, the use of SCMs often causes longer setting times [10], lower early strength development [5] and an increase in drying shrinkage [9,11].

Several methodologies have been studied to overcome the drawbacks and improve the efficiency of OPC and SCMs in cementitious systems which include three main methods: thermal, mechanical, and chemical. The heat treatment i.e. thermal activation can be divided into calcination [12–14] and elevated temperature curing [15]. The former has been found highly contingent on limiting factors including the reactivity of the amorphous phases and also the decrease of specific surface area and soluble fraction and the increase of crystalline fraction. The latter often causes lower strength development in cementitious materials incorporating SCMs at later ages [16,17].

Mechanical methods have been extensively utilised to enhance the pozzolanic activity of some types of SCMs by grinding them into ultrafine powders for a prolonged period of time. This decreases the particle size distribution and increases the dissolution rate of pozzolans, which accelerates the pozzolanic reaction rate and consequently the strength development of concrete containing SCMs. However, controversial results are also obtained regarding the correlation between the fixed free portlandite (CH) and the pozzolans' surface area as well as insignificant ultimate strength improvement [16–19]. Moreover, the more fine the particles, the more chance of aggregation and agglomeration due to the interaction forces between particles inside the matrix.

The utilisation of chemical accelerators (like $CaCl_2$) to accelerate the early-age cement hydration has been investigated. The chloride ions, however, adversely interfere with various hydration products and could also cause depassivation of steel in reinforced concrete triggering corrosion and cracking of concrete [20]. Other types of chemicals have also been identified that have shortcomings for long-term strength and durability of cement-based composites [21,22]. Surface treatment of GGBS with chemical activator(s) [23] changes the properties of their surface layer, which accelerates the rate of pozzolanic reactions, enhances strength development rate, and increases the ultimate strength of cement-based composites. Nevertheless, some of the most effective chemicals, such as caustic alkalis are not cost effective, user/eco-friendly or are not practically feasible [24].

Furthermore, concretes subjected to high temperature curing at early ages attain higher early-age compressive and splitting tensile strengths but significantly lower long-term strengths and elastic modulus than those subjected to normal temperatures [25]. Whilst the addition of heterogenous nucleation sites (with added synthetic C-S-H particles or limestone) were also found to be effective on cement hydration acceleration [26], this approach is still very expensive and practically limited.

Power Ultrasound has been used over a wide range of applications in materials science, from surface cleaning and degassing, to particle dispersion and the production of nanostructures. Although some of these applications have been investigated extensively, others, such as the use of PUS to control the properties and performance of cementitious materials and enhancing the properties of SCMs incorporating composites are yet to be reviewed and hold new, exciting possibilities. Several ultrasound techniques have been used to characterise the setting and hardening processes of cement pastes, mortar and concrete, providing an overall evaluation of mechanical strength, porosity, permeability and durability of cementitious composites [27]. A few studies have focused on the effect of power ultrasound on the performance of early age hydration reactions in cementitious matrices as well as composites incorporating SCMs. In this paper, a review of the literature on the effects of PUS on the early age hydration kinetics of PC and binary blends, together with some underlying mechanisms involved is presented. The future for applications of PUS in cement-based materials will also be considered.

2. Ultrasonication

2.1. Background

The first reports of the chemical and biological effects of ultrasound were published in the early 20th century [28–30]. However, it took many years for industry to adopt ultrasound as a more general energy source to drive a range of chemical and processing operations. In 1972, Neppiras reviewed the main applications of power ultrasound (PUS) in industry; which at that time was referred to as macrosonics [31]. The major applications lay in cleaning, plastic and metal welding, wire and tube-drawing, ultrasonic machining, teeth descaling and the extraction of chemicals from plants. He also listed some "minor applications" which have since become rather more important. These included shaking and sieving [32], electrolytic processes [33], crystallisation [34], emulsification [35], dispersion [36], depolymerisation [37], degassing, the production of aerosols, drying, defoaming and sterilization [38].

The various strands of the applications of power ultrasound in the chemistry and material processing fields were later to be brought together under the umbrella title of sonochemistry. The first ever international conference on sonochemistry was held at Warwick University in the UK in April 1986 and sonochemistry was further established as a subject in its own right with the publication of two major reviews [39,40].

2.2. Conventional power ultrasound

Conventional power ultrasound (PUS) is used in the frequency range between 20 kHz and 100 kHz (Fig. 1) and is able to generate acoustic cavitation in a liquid and cavitation is the driving force for sonochemical reactions. PUS is generally at a lower frequency range than that used for diagnostic ultrasound which is in the MHz range.

The most common way of generating ultrasound is by exposing a piezoelectric ceramic transducer to a high frequency alternating electric current. Under the influence of this electrical field the piezoelectric material expands and contracts producing a high frequency mechanical vibration. This vibrational motion can be transferred into and through any liquid medium, inducing cavitation and associated physical and chemicals effects [41–43].

2.3. Acoustic cavitation phenomena and cement-based materials

The chemical and physical effects of PUS in a liquid medium have been universally acknowledged to be the consequence of acoustic

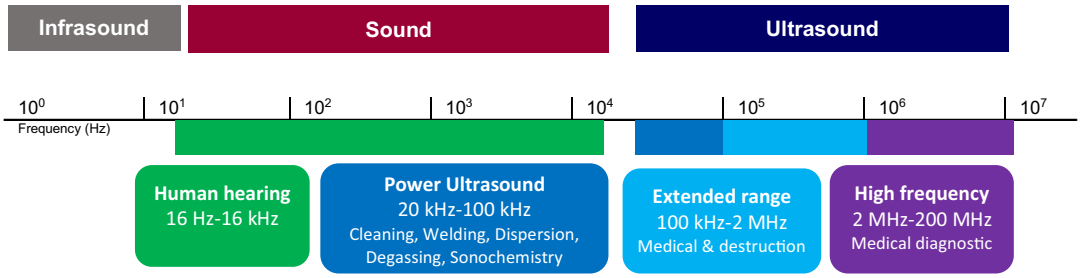


Fig. 1. Illustration of frequency spectrum from infrasound to ultrasound.

cavitation. This is the formation, growth, and collapse of gaseous microbubbles in the liquid phase (Fig. 2). Ultrasound is transmitted through a material in the same way as any sound wave via a series of compression and rarefaction cycles. During rarefaction, provided that the negative pressure is strong enough to overcome the intermolecular forces binding the fluid, the fluid is literally torn apart producing tiny cavities (microbubbles) throughout the medium. In the succeeding compression cycle if cavities were enclosing a vacuum, they would collapse almost instantaneously. However, during cavity formation a small amount of gas or vapour is drawn in from the surrounding liquid. As a result, the succeeding compression cycle may not totally collapse the bubbles and so they will grow slightly larger in the next rarefaction cycle with a further intake of gas and vapour. The process is known as rectified diffusion. The bubble will not grow indefinitely, there will be an equilibrium size for any bubble in an acoustic field (this depends on frequency). Some bubbles will continue to resonate in this stable state but many will become unstable and collapse, generating microspots of extreme conditions of temperature and pressure. Based on the theory which has been put forward to explain the energy release involved with cavitation, each cavitation bubble acts as a localised microreactor which generates instantaneous temperatures and pressures on collapse of several thousand degrees and over one thousand atmospheres respectively [41]. This phenomenon has led to the most popular and widely accepted theory regarding the explanation of the effects of cavitation collapse the "hot spot" theory [44]. The implosion of cavitation bubbles results in the formation of "shock waves" (strong pressure waves) and "jet streams" inducing a "microstreaming" effect, causing jets of liquid to be directed to the solid surface of any material suspended in the liquid and resulting in particle size reduction, particle collisions and surface activation/cleaning. The release of shock waves and the effect of microstreaming together may also cause intensive shear forces inside the liquid medium [43]. It is these effects which make PUS attractive to the cement-based materials industry. Here, the

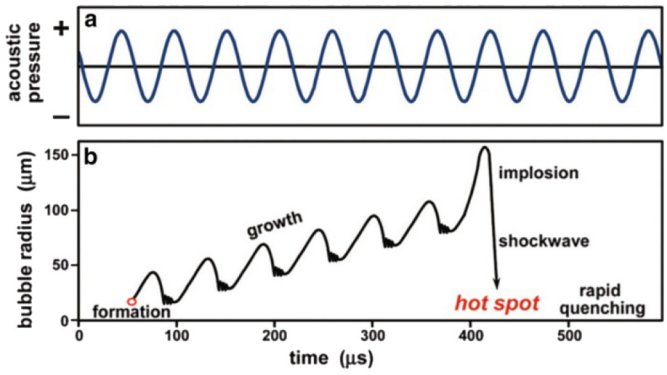


Fig. 2. Schematic illustration of the process of acoustic cavitation: the formation, growth and implosive collapse of bubbles in a liquid irradiated with high intensity ultrasound. Reproduced courtesy of Xu et al. [45].

systems are heterogeneous and material hydration is important during the transformation of a fluid suspension into a solid at room temperature with minimal bulk volume change.

3. Mechanisms of Portland cement hydration

Many modelling and experimental works explain the phenomena occurring during cement clicker hydration and that of blended systems. It is universally hypothesised that the Portland cement hydration in which transformation of anhydrous to hydrate phases occurs is basically a dissolution-precipitation process [46]. It has been found that due to the congruently charge balanced dissolution of all the anhydrous phases, crystal structure would preclude the unrestricted dissolution of certain phases without others. So, during the hydration progress, the potential hydration products would intrinsically have a higher stability than the anhydrous phases [46–48].

The hydration of tricalcium silicate (C_3S) referred to as alite, as the most important constituent of Portland cement clinker (accounting for 50–70% by mass) has been extensively investigated. Since its reaction kinetics mostly resemble those of Portland cement, alite is being considered to serve as a reliable model in studying Portland cement hydration [49]. The principal product of alite hydration is calcium-silicate-hydrates (C-S-H), a nearly amorphous phase, which primarily contributes to the strength and volume stability in cementitious materials [50]. Classical theories of hydration indicated that the surface of cement grains begins to be covered by nuclei of the C-S-H gel formed and grown within a few seconds after initially mixing with water. Remaining stable only in the induction period (which occurs because the size and number of growing regions are small); this impermeable metastable C-S-H phase has a greater solubility than C-S-H and a lower solubility than alite. Other researchers found two types of C-S-H during the initial stages of hydration including low density C-S-H and high density C-S-H [50,51]. However, according to the most leading theory known as the nucleation theory, it has been postulated that the induction period is controlled by the changes in ion concentration in the solution rather than the presence of inhibiting hydration layers formed on the surface of alite grains. Once the alite grains become covered with hydration

Table 1
Identified phenomena controlling early hydration kinetics, summarized from [52,54].

Mechanism	Overall kinetic behaviour	Reaction stage
Dissolution	dissolving alite grain and releasing ions into the pore solution	Up to the end of the induction period
Diffusion	transport of solution components through the pore volume of cement paste	
Nucleation	initiator of precipitation of solids heterogeneously on solid surface or homogenously in solution	Up to main heat evolution peak
Growth	surface attachment, incorporation of molecular units into the structure of crystalline or amorphous solids	

products, the rate of hydration becomes controlled by the rate of diffusion through this layer and decreases slowly for weeks or months. The theory proposed that the hydration of alite is mostly controlled by solution controlled dissolution/diffusion and nucleation with densifying growth mechanisms (Table 1). According to the nucleation theory, the rate of hydration during the induction period is dominated by nucleation and growth of C-S-H formed at the early stage and this period ends when the growth of C-S-H starts. During early stages of hydration, the primary hydrates precipitate on the surface of alite, but not as a continuous membrane (Fig. 3) [46,51–54].

It has also been postulated that the nucleation/growth mechanism might be a continuous process, implying that existing C-S-H nanostructures stimulate the formation and expansion of new products outward from the original nucleation site [53]. Overall, it seems that crystallisation plays a significant role in cement-based materials hydration, controlling early and long-term properties of cementitious composites.

4. Effect of PUS on crystallisation of inorganic materials

Power ultrasound has proved to be extremely useful in crystallisation processes. It serves a number of roles in the initiation of seeding and subsequent crystal formation and growth. This may be due to cavitation bubbles acting as nuclei for crystal growth or by the disruption of seeds/nuclei already present within the medium thus increasing the number of nuclei present in the solution. These effects have been examined in cementitious system by the addition of synthetic C-S-H [26] or nanomaterials [56]. The Insonation of gels formed from sodium aluminate and sodium silicate leads to increases in the nucleation and crystallisation rates for the formation of zeolites by up to 6-fold and 3-fold respectively at 85 °C [57]. The zeolite formed in the ultrasonic field shows reduced particle size and a narrower size distribution compared with those produced by conventional methodology. It was suggested that the reduction in particle size was due to an increase in the number of crystallisation nuclei and their dispersion by the acoustic field. The rate of nucleation was shown to increase with increasing irradiation intensity and this was also accompanied by a reduction in particle size [57,58].

One of the most relevant research findings relating to the effects of PUS on crystallisation involving cementitious materials is found in the studies carried out on the precipitation of calcium carbonate by Nishida [59]. He examined the influence of ultrasonic irradiation on the precipitation rate of a supersaturated solution of calcium carbonate ($CaCO_3$). He observed that PUS accelerated the precipitation of $CaCO_3$. He identified macrostreaming as a dominant factor of this phenomena rather than microstreaming which is generally known as one of major physical effects of cavitation. In a newer study, Boels et al. [60] investigated the seeded crystallisation of calcite affected by the PUS. The measurement of volumetric crystal growth rate of calcite seed crystals showed that the ultrasonic irradiation considerably increased the crystallisation rate and surface area available for crystal growth and also caused disruption of conglomerates of single crystals.

Other experiments were carried out on salts like Barium sulphate ($BaSO_4$) [61,62] and Potassium sulphate (K_2SO_4) [63]. Guo et al. [61,62] studied the effect of ultrasound on crystallisation and the homogeneous nucleation of $BaSO_4$ and concluded that the induction time significantly decreased with increasing PUS energy due to the accelerated diffusion process. Likewise, the effect of ultrasound on primary nucleation of K_2SO_4 investigated by Lyczko et al. [63] showed a significant reduction in induction time and accordingly the nucleation mechanism enhancement. Promotion of the dissolution-precipitation of the aluminium hydroxide-water system affected by the moderate sonication was documented by Enomoto et al. [64]. A study [65] of the influence of PUS in controlling the supersaturation, nucleation and crystal growth during the acid–base reaction crystallisation showed the effectiveness of mixing, deagglomeration, reduction of both the induction period and metastable zone width of the crystallisation and also the modulation of the crystal size and size distribution. Similarly, the potential of PUS on controlling the crystallisation process of a type of antibiotics (roxithromycin) through the inducing accelerated nucleation, reduction of the induction time, suppressing agglomeration and changing crystal habit were investigated by Gou et al. [66].

Overall, the studies completed on the effect of PUS on the precipitation of inorganic materials generally suggest that ultrasonication provides enhanced dissolution-precipitation, and consequently more nucleation sites and surface area for a more efficient growth of crystals. Therefore, this may be a promising approach for promoting the

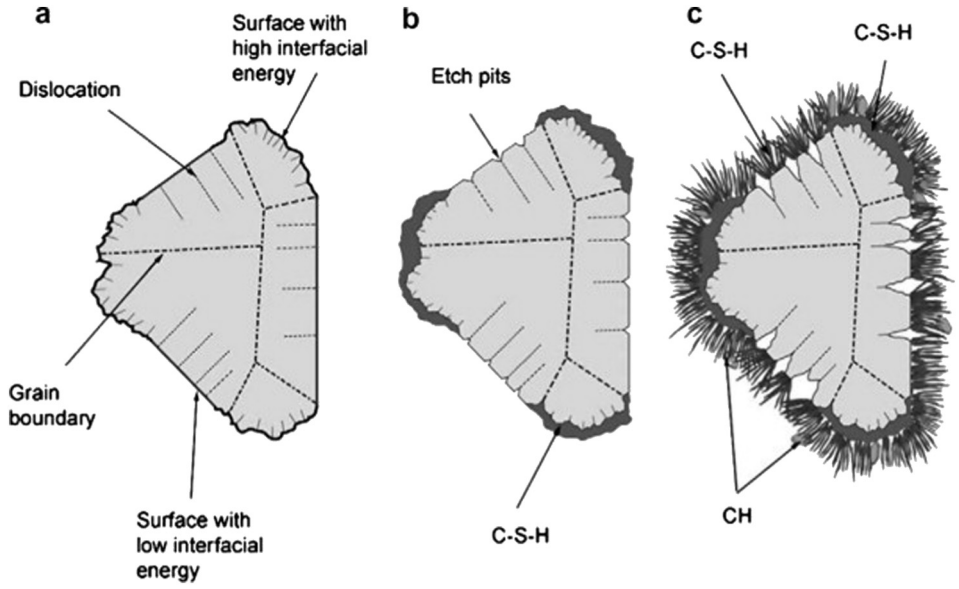


Fig. 3. Schematic representation of alite grain hydration at early stages (a) an anhydrous alite grain (b) the alite grain surface subjected to water with C-S-H formed, along with etch pits (dissolution voids) formation (c) finally stable nuclei of C-S-H and CH initiate growing, Reprinted courtesy of Juilland et al. [55].

crystallisation properties of hydration products in cementitious materials.

5. Influence of PUS on cementitious systems

5.1. Challenges in cement-based systems

It has been recognised that aggregation or agglomeration can be induced between particles due to the interaction forces between particles of colloidal/suspension systems (including the electrical double layer, van der Waals, Born, hydration and steric forces) [67]. During these processes, primary particles stick to each other, and spontaneously form irregular particle clusters, flocs, or aggregates held together by these weak forces that can be separated. So, large irregular agglomerates embedded in cement-based composites can significantly reduce the effectiveness of clinker cement and SCMs to participate in hydration as well as the pozzolanic reaction and consequently improve all those positive properties which pozzolans can bring to them. In terms of microstructural chemical reactions, the presence of SCM agglomerates leads to the formation of a C-S-H with a much higher calcium to silicate (Ca/Si) molar ratio than usual in cementitious composites [68,69]. Generally, in cement chemistry and concrete technology, there are several challenges associated with hydration of Portland cement and SCMs particles which might be approached by PUS application:

- Aggregation/agglomeration of cementitious materials; controlling packing density and hydration rate.
- Pore structure of hardened cement-based materials which significantly affects the mechanical properties and durability [70].
- Penetration/diffusion (dissolution) of anhydrous phases in the fluid suspension of cementitious materials during the hydration process occurs at the rapid slow down in reaction stage at the beginning of hydration leading to a period of slow reaction known as induction time and continues through the growth process after the acceleration period; controlling early age and long term properties of cementitious materials [46,50].
- Hydration rate after acceleration period (growth of hydration products) appears to be even slower compared to crystalline hydrates, most probably due to the lack of available surface area and space for hydration; controlling the long-term properties of cementitious materials [46].
- Hydration products can act as deposit/cover on the cement/SCMs particles and hinder further reactions [46].
- Complex hydration kinetics; this appears to make each of the phases hydrate at a rate different from the others and are also affected in presence of each other [46].

5.2. Postulated mechanisms concerning the phenomenon

It is thought that the most promising effects of PUS will relate to the homogenising and dispersion of cementitious particles through a colloidal cement system, leading to the deagglomeration of particle clusters. This will enhance the effectiveness of SCMs in the secondary hydration reactions. The intensity of cavitational effects which depends on the size and type of material presented in the medium, can lead to mechanical disaggregation and dispersion of loosely held clusters, the removal of surface coatings by abrasion, and enhance mass transfer to the surface.

Power ultrasound might also be influential on the crystallisation process of different hydration phases in a cementitious system. Furthermore, exfoliation of Portland cement and the anhydrous and hydrated phases of SCMs through the generation of surface damages triggered by the effects of cavitation bubble collapse might control the rate of hydration products and ultimately the properties of cementitious composites. The key mechanical effects of PUS offer an opportunity to overcome the types of challenges associated with cementitious materials outlined in the Table 2.

In recent years, the application of ultrasonic treatment to disperse different types of powder and nanoparticles in an aqueous and non-aqueous solution as well as high viscosity polymer solution has been explored [36]. Despite the limited efforts to characterise the ultrasonic dispersion of densified silica fume and apply it to cementitious systems (as well as one study carried out concerning the characterisation of the PUS effect on hydration and fluidity of cement/slag blended suspension), still relatively little is known about the effects of PUS on Portland

Table 2
Proposed physical/mechanical effects of ultrasound cavitation tailored for cementitious materials.

Systems	Postulated/investigated effects of PUS	Expected results in cementitious materials
Homogenous (in absence of pre-existing crystal; liquid bulk)	Enhanced mass and heat transfer from and onto the surface due to microstreaming [34,43,71]	Intensification/accelerated transportation of anhydrous cementitious materials and hydration phases/products
	Degassing of suspension [43]	Highly critical to pore structure of cement-based materials systems mainly air entrapped voids and air entrained voids (generated by air entrained agents) controlling both mechanical and transport properties
	Enhancing crystallisation/precipitation (technically known as "sonocrystallisation") [34]	Reduction of the induction time Enhancing homogenous nucleation and growth of stable hydration products Reduction of metastable zone width
Heterogeneous (in presence of wall or existing crystals; liquid-solid solution)	Breaking up/erosion of the surface structure (surface damages) due to shock waves and/or jet streams [34,43,72]	Modifying the morphology of crystals/hydration products Allow penetration of phases entered in hydration reaction by affecting etch pits in the surface Release of hydration products from cementitious materials surfaces Allow penetration of particles already covered with hydration products by removal/release of formed hydration precipitates away
	Degradation of large solid particles due to shear forces induced by shock waves and microstreaming [34,41,43,72,73]	Reduction of particle size and agglomeration Increase surface area of cement particles and SCMs Homogenising and Dispersion of anhydrous cementitious particles and hydrated phases
	High velocity collisions and accelerated motion of particles [34,43,74]	More effective mass transfer, consequently intensification of those effects occur in homogenous systems
	Enhancing crystallisation/precipitation [34]	Reduction of the induction time Enhancing heterogeneous nucleation and growth of stable hydration products Reduction of metastable zone width

cement and SCMs incorporated in cementitious systems. This review will focus on research carried out on the capability of PUS to improve the properties of cementitious materials. First, a discussion will be made on the potential beneficial effects of cavitation on cementitious materials, followed by some comments on the limited number of results published related to practical applications of PUS to Portland cement and SCMs.

5.3. Parameters influencing the cavitation phenomena in cement-based materials

It has been found that some external parameters have a great influence on cavitation as the most important phenomenon to induce sonochemical effects. Understanding these factors helps to provide condition in which the sonochemical effects are optimised. Some of the most influential factors affecting the sonochemical effects with regards to cement-based system are reviewed in Table 3.

Table 3
Review of some factors influencing the cavitation phenomena in cement-based materials.

Parameters	Sonochemistry perspective	Cementitious materials perspective
Frequency	At higher frequencies, the rarefaction/compression cycle becomes shorter than the time required to permit the molecules to be pulled apart, making the cavitation difficult or impossible to achieve. Moreover, for a determined power level, the threshold in changing physical effects might be reached and so no further changes could be observed as a result of sonication. Frequencies usually opted for typical crystallisation fall in the range between 15 and 40 kHz which its variable was found to have the same influence on nucleation and growth [28]	initiation of acoustic cavitation using combined low and high-frequency which might have substantial effects on the hydration crystallisation of cementitious materials.
Intensity (Power Input)	Whilst an increase in ultrasound intensity will provide an increase in the sonochemical effects, the barrier effect of larger and longer lived bubble formation to the transfer of acoustic energy, possibility of transducer material fracture and great loss in efficiency of power transfer from the source as a result of decoupling phenomena need to be considered [28,34].	An increase in ultrasound power intensity is expected to cause heavier flow, enhanced mass transfer in the media and the driving force of crystallisation, leading to the higher the crystallisation rate.
Temperature	Apart from a decrease in viscosity and surface tension, any increase in temperature will raise the vapour pressure of a medium and therefore will cause an easier cavitation with a less violent collapse. However, at higher temperatures, a large number of cavitation bubbles are generated because of the approaching solvent boiling point. Acting as a barrier to sound waves transmission, these will cushion the ultrasonic energy from the source	In the cementitious systems, the intensive increase and decrease in temperature will markedly accelerate and slow down the hydration rates, leading to a relatively fast and slow initial setting times, respectively, and adversely affected microstructure and hardening properties. Therefore, practical and technical considerations need to be taken when applying the PUS.
Gas bubbles	Dissolved gas and small gas bubbles in a fluid act as nuclei for cavitation, promoting cavitation and are then removed.	Degassing and defoaming characterisation of PUS is expected to be highly critical as the products of the hydration process consist of a pore system governing the properties of cement-based systems. The effect of PUS on the stability of entrained air bubbles has also needs to be understood.

6. Studies on the influence of PUS on cementitious materials

6.1. Portland cement

An experimental study performed by Rößler [75] and Peters [76] represents the first officially published experiment carried out on the influence of PUS on initial setting, hardening and hydration characterisation of Portland cement (CEM I 42.4R). In order to guarantee a uniformly continuous sonication until providing the required specific energy input, a flow-through cell sonication set-up equipped with a laboratory ultrasonic horn was utilised (UIP 1000hd, Hielscher, Germany). The set-up provided a constant frequency of 20 kHz and different amplitudes adjustable by a booster.

In the first part of the study, the research aimed to highlight the effect of PUS on setting and hardening of a cement suspension (with a w/c ratio of 0.37). The practical limitations regarding the use of PUS in concrete production, namely the initial setting time, temperature rise due to the ultrasound application and the minimal period of PUS treatment were considered. So, the optimal PUS parameters emerged to be 43 μm (ultrasonic horn with front face diameter of 0.9 cm^2) and 75 J/ml for the PUS amplitude and specific energy input, respectively. The results of initial setting time demonstrated that ultrasound treatment shifts the initial setting time of cement paste to an earlier time. Moreover, the influence of PUS on cement hydration was evaluated by means of analysing the isothermal differential conduction calorimetry and the non-destructive ultrasonic P-wave velocity and microstructural study of cement suspension as well as the compressive strength development of mortars [76].

The results of isothermal heat release demonstrated that PUS accelerates the heat release rate limited to the hydration acceleration period, whereas the total heat release was unaffected by the PUS treatment (Fig. 4). Relying on the correlation between the P-wave velocity test results and the strength development of cementitious materials; it was deduced that an improved strength development of sonicated cement suspension limited to only the first 16 h was most probably due to the accelerated cement hydration [76].

The enhanced compressive strength of mortars during the 24 h of hydration as well as microstructure analysis of cement suspension (Fig. 5) underscored the PUS effectiveness on accelerating the cement hydration, confirming the results obtained from the hydration heat release and setting time examination [76].

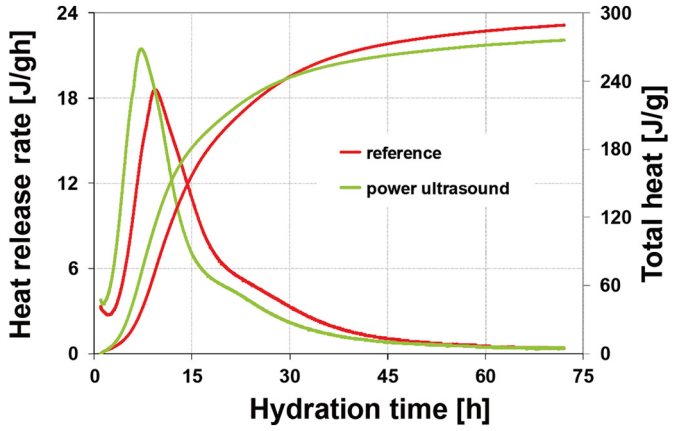
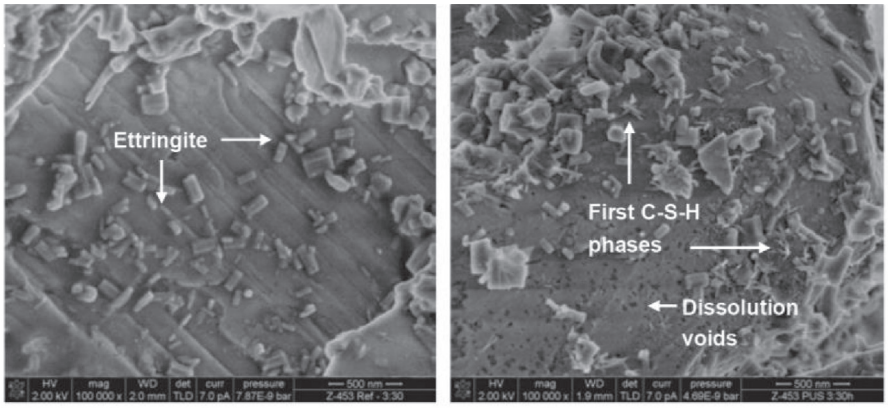
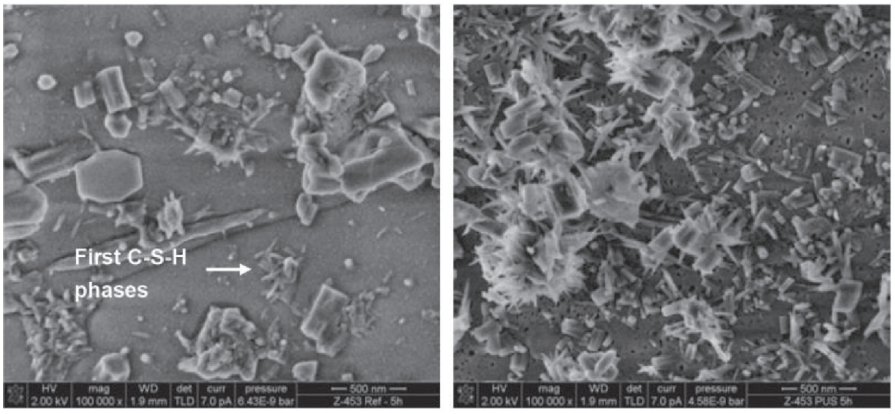


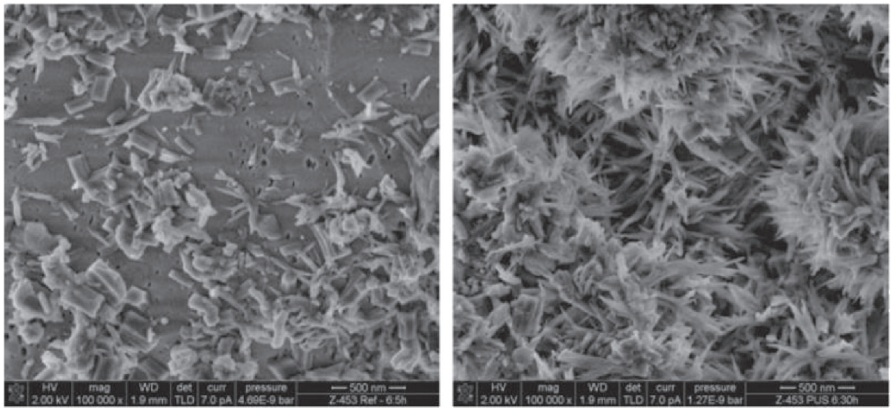
Fig. 4. Isothermal heat release rate and total heat of reference and sonicated cement suspension (w/c: 0.37 with 0.1 wt% SP) during first 72 h of hydration, Reprinted courtesy of Peters [76].



(a) 3 hours and 30 minutes hydration



(b) 5 hours hydration



(c) 6 hours and 30 minutes hydration

Fig. 5. SEM micrographs of reference (left) and PUS treated (right) cement suspension microstructure (w/c: 0.37 with 0.1 wt% SP), Reprinted courtesy of Peters [76].

Furthermore, in-depth investigations were performed on the characterisation of model substance hydration (synthesised alite) in order to evaluate primary (homogenous) and heterogeneous crystallisation and precipitation (nucleation and growth) of C-S-H phases. The former examination was conducted on clear Ca-Si solution and the latter was performed on diluted alite suspension. The results of electrical conductivity measurement and determination of Ca and Si ion concentration by the ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) showed that PUS accelerates the kinetics of alite hydration but does not alter the reaction path. It was hypothesised that the mechanism of hydration acceleration facilitated by the ultrasound introduction would be dominated by the precipitation enhancement of very early C-S-H phases. Besides, it was postulated that the shock waves induced by the cavitation phenomenon is mostly responsible for increasing the particle collisions, leading to localised erosion effects and the removal of initial C-S-H precipitates away from alite surfaces.

Consequently, not only do the alite grain surfaces remain unwrapped with C-S-H products, but also detached C-S-H precipitates provide additional sites for triggering more C-S-H phases growth. It was proposed that other PUS effects like hot spot and jet streams were accounted for a minor significance [76].

The positive influence of PUS on fluidity and the generation of homogenous fresh cement paste was also confirmed by Peters by performing mini slump flow spreading time and V-funnel flow time examinations. Further observation was carried out to briefly characterise the hydration process of GGBS under the influence of PUS. Acceleration of GGBS blended cement hydration was deduced based on the initial setting time, compressive strength evaluation of mortars and isothermal heat release rate examination. Nevertheless, the isothermal heat calorimetry test on pure GGBS suspension incorporating CH showed that PUS application only slightly accelerates GGBS [76].

Recently, Resonance Acoustic mixing (RAM) technology that combines the principals of reciprocating movement agitator mixing and bubble acoustic micro-streaming mixing was employed by Vandenberg and Wille [77] to evaluate its efficiency for mixing Ultra-high performance mortar. The results presented reduced flow and workability properties whilst improved mechanical properties in specimens mixed with RAM. Although an increase in hydration kinetics and change microstructural development of mixtures compared to a standard table paddle mixer were concluded, the study was not engaged to perform microstructural study and quantitative reasoning.

Overall, although some test results confirm the positive influence of PUS on cement hydration, some other examination results are not reflective of an entirely significant effect of hydration enhancement. More research, especially quantification of hydration is needed to gain a fundamental understanding about the mechanisms in which PUS affects cementitious systems.

6.2. SCMs

The mechanical effects of cavitation are already being used in generation of nanostructures and nanoparticles [36]. These physical effects also have been postulated to provide a reduction in the size of densified or agglomerated particles by applying the forces of cavitation, breaking of agglomerates and aggregates. In this section, the studies performed to characterise the effects of PUS on SCMs have been overviewed.

It has been reported that silica fume as a SCM will significantly reduce both the interfacial transition zone between the aggregates and bulk cement paste and the deposition of a portlandite rim, resulting in improvement in fresh properties, mechanical strength and durability properties in cement-based materials [78–81]. However, the continuous interaction of individual spheres of silica fume particles promotes the formation of agglomerates. As in most silica-fume-bearing cementitious composites, the silica fume is introduced in its densified (agglomerated) form, and the agglomerates are likely to remain at least partly undispersed especially in normal concrete mixing [69,82]. This inhibits the complete transformation of silica rich gel into different morphological types of C-S-H through the pozzolanic reaction and consequently leaves a large number of unhydrated or partly reacted remnants of silica fume particles in the hardened cement paste [83]. As for all SCMs, the un-reacted silica fume particles might reduce the effectiveness of pozzolanic reactions and adversely affect the positive contribution of them for improving mechanical and durability properties of cement-based composites [84].

Some studies (Table 4) have been carried out to deal with the dispersion of silica fume by utilising the PUS approach, as an alternative to enhance its pozzolanic activity. Overall, it has been inferred that the sonication process generally shifts the particle size distribution towards smaller particle sizes, comparatively raising the volume content of sub-micrometric particles in the PUS treated silica fume suspension.

Gapinski and Scanlon [82] recommended a sonication process to break and disperse the bonded silica fume agglomerates.

Martinez-Velandia et al. [85] studied the granulometric properties and deagglomeration of the different types of silica fume affected by PUS. The study confirmed the effectiveness of the PUS treatment on the granulometric distribution [85].

In order to investigate the effectiveness of sonicated silica fume particles on the pozzolanic reactivity in cementitious system, Martinez-Velandia et al. [84] Introduced their optimised homogenous suspensions of silica fume treated by the means of the sonication process [85] to cement mortars. Sonicated silica fume incorporated mortars showed an increase of 10 to 15% in the compressive strength especially for longer sonication times and higher sonication power levels. The thermogravimetric study also confirmed the activation of the pozzolanic reactions (fixations of free portlandite) [84].

Rodriguez et al. [68] found that sonication treatment introduced to densified silica fume contributes to an increase in the volume of particles of smaller sizes and also the enhancement of its reactivity by a de-agglomeration mechanism, improving dispersion.

The effect of sonicated densified silica fume on the hydration products, performance and pore structures of blended cement based pastes was the topic for another study carried out by Rodriguez et al. [86]. The results of the compressive strength development study showed that applying the sonicated silica fume promotes higher mechanical strength in cement mortar specimens so that a desirable mechanical performance can be achieved using a smaller content of SCMs. Moreover, the results of XRD, TGA/DTG analysis indicated that PUS treated silica fume shows higher pozzolanic reactivity, associated with higher consumption of portlandite through the hydration process compared with the densified silica fume admixture. Based on the results of MAS-NMR spectroscopy analysis, this causes the formation of an aluminium-rich C-S-H with a structure of longer chain length and lower Ca/Si ratio which is a more stable binding phase. The pore size distribution study of cement mortar specimens also showed that the incorporation of sonicated silica fume induces a significant decrease by more than 20% in the volume of large pores (with diameters larger than 10 μm) as well as a reduction in total porosity.

Hashem et al. [87] studied the sonication process parameters for the preparation of nanoparticles of silica fume and used the optimum sample to investigate the pozzolanic reactivity by introducing it to a concrete mixture. Based on the results obtained, the optimum sub-nanometric particle distributions of ultrasound treated silica fume correspond to the experimental condition: sonication power/frequency/process time: 135 watts/40 kHz/60 min; homogenizer speed of 24,000 rpm and liquid/water ratio of 6. Introducing this optimised sample into cementitious system by 1% cement replacement; the sonicated silica fume incorporated concrete showed 10 and 20% enhancement in compressive strength compared to the reference silica fume and control mixtures, respectively. Sharobim et al. [88] found that sonicated nano-silica enhances compressive strength of concrete and the microstructure of the cement matrix. As it is evident, all the reviewed studies have investigated the dispersion improvement of silica fume and nano-silica in the cementitious matrix whilst failing to address the kinetics of cement-based materials hydration under the effect of ultrasound.

Askarinejad et al. [89] characterised the natural pozzolans' nanostructures affected by introducing the PUS irradiation to bulk natural pozzolans by SEM and XRD as well as studying the pozzolanic activity by TG/DTA analysis and mortar's compressive strength index. Similar to the results obtained from the studies carried out on the effect of PUS on densified silica fume, the results demonstrated that the pozzolanic reactivity of natural pozzolans was enhanced by applying PUS in optimised conditions.

Table 4
Summary of PUS studies on silica fume.

Author, year	Testing materials	Sonication approach	Test equipment & conditions	Characterisation approach
Gapinski & Scanlon; 2006 [82]	Densified silica fume (as received sample)	Built in 600 watt ultrasound	0.010% sodium pyro-phosphate solution as the surfactant	custom-built laser scattering particle size analyser with and a
Martinez-Velandia et al.; 2008 [85]	3 types of densified silica fume with different granulometric distribution 1 type No-densified silica fume 1 type of Milled densified silica fume	Sonicator S-3000, Misonix Max. 600 watts; 20 kHz Different sonication power: 141 (2.26 watt/g) and 168 (2.69 watt/g) (corresponding to 80 and 100% of the maximum power of sonication probe) Different sonication time: 2, 4, 6, 8, 10, 12, 14, 16, 18, and 20 min.	600 ml precipitate beaker suspended in water Different SF/water ratio Temperature: 20–35 °C	Granulometric distribution curves analysis by calculation of mean particle diameters, 10, 50, and 90% percentiles and the volume of particles with a diameter lower than 1 μm (Submicrometric particles) Laser diffraction granulometric (LDG) analysis (Mastersizer 2000, Malvern Instruments); Specific surface area (SSA) measurement by BET method (Tristar 3000, Micromeritics)
Martinez-Velandia et al.; 2011 [84]	1 type of densified silica fume (supplied by Ferroatlantica, Sabon, Spain) Sonicated silica fume by the procedure stated on the previous work [85] Cement: CEM I-52.5R Superplasticizer Melment 240 (Bettor S.L)	Sonicator S-3000, Misonix Max. 600 watts; 20 kHz Different sonication power, watts (watt/g): 60 (0.96), 81 (1.30), 111 (1.78), and 141 (2.26) Different sonication time: 5, 10, 15, 20, 25 min.	Preparation of cement mortars according to EN 196-1:1996 procedure: 5, 10 and 15% cement replacement by silica fume; Water/cm: 0.35; Curing time: 7 and 28 days 600 ml precipitate beakers suspended in water (228.4 ml) Different SF/water ratio Temperature: 20–35 °C Mortars was ground in acetone and dried for 30 min at 60 °C	Laser diffraction granulometric (LDG) analysis SEM micrographs (JEOL JSM 6300) Calculation of the strength activity index (SAI) Study of fixation of hydrated lime by: TGA/DTG analysis (TGA Mettler-Toledo 850 thermobalance, 35–600 °C at 10 °C/min)
Rodriguez et al.; 2012 [68]	3 commercial densified silica fume 2 non-densified silica fume with different particle size distribution Cement paste: CEM-I 52.5R, silica fume sonicated suspension, Cement replacement 10%	Sonicator S-3000, Misonix Max. 600 watts; Output frequency: 20 kHz Sonication time: 10 min	Aqueous dispersion: 5 g of SF and 20 g of water in a precipitate beaker of 50 ml water/binder ratio: 0.4 Temperature kept steady at maximum 40 °C using an external cooling bath	PUS treated particles characterisation by SEM micrographs, XRD analysis, laser diffraction analysis (DLA), zeta potential measurement and TEM micrographs, Microstructural characterisation of cement paste samples affected by sonication treatment by TGA/DTG and SEM/EDS analyses
Rodriguez et al.; 2012 [86]	1 commercial densified silica fume (supplied by Elkem Silicon Materials, 940D) Cement: CEM I-52.5R	Sonicator S-3000, Misonix Max. 600 watts; Output frequency: 20 kHz Sonication time: 15 min	Aqueous dispersion: solid to liquid ratio of 0.20 Temperature kept steady at maximum 40 °C using an external cooling bath 40 mm Cubic samples of mortar containing blended cements with 5, 10 and 20% cement replacement by densified and sonicated silica fume admixtures Water to binder ratio: 0.3/Superplasticizer (Glenium ACE31, BASF)	PUS treated particles characterisation by TEM micrographs Paste characterisation on blended cement pastes with w/b:0.5 and 10 wt% substitution of silica fume for cement by XRD, TGA/DTG and MAS-NMR analysis Compressive strength development test (7, 28 and 60 days of curing) Pore size distribution of mortar specimens (28 days) by MIP (AutoPore IV 9500, MIC) Microstructural characterisation by SEM/EDS analyses
Hashem et al.; 2015 [87]	Commercial ultra-fine silica fume with particle size ranging from 625 nm to 48 nm Concrete mixture: 65% coarse aggregates and 35% fine aggregate Polycarboxylate admixture (Glenium C315 SCC)	Ultrasonication Bath (FALC Instruments) 150 watts, Frequencies 40 and 59 kHz Power set on 70 watts (according to 50% power) and 135 watts (100% power) Frequency set to 59 kHz Different sonication time: 7, 15, 60, 150 min.	600 ml precipitate beaker Different liquid/powder ratio: 1.5, 2.4, 6, 10 and 12 Mechanical homogenizer (rotor-stator) with working speeds of 9500, 13,500 and 24,000 rpm Temperature kept steady at maximum 40 °C using built-in heater Concrete mixtures, by 1, 5 and 10% untreated silica fume and 1% treated silica fume	Particle size analysis, calculating mean. Range, D50, D80, D20 and sub micrometre sizes. TEM micrographs of treated particles (JEOL JEM-1230) Zeta potential measurement (Zeta Sizer 2000, Malvern Instruments) Compressive strength measurement of concrete

Overall, It is evident that ultrasonic treatment has been shown to be a favourable alternative in enhancing the efficiency of densified silica fume and natural pozzolans in the following forms: improved dispersion of agglomerates (especially sub-micrometre particles leading to a much wider particle distribution and enhancing the pozzolanic activity associated with a lower amount of unreacted particles), and comparative mechanical performance using a lower dosage of SCMs replacing part of Portland cement. However, very little is known about the PUS influences on SCMs and extensive specific research is needed to clarify the mechanisms in which PUS influences Portland cement and SCMs hydration.

7. Conclusion and future perspectives

This review has surveyed existing research in the field of PUS applications in cementitious materials. In doing so, some specific research areas have been identified which are needed to make progress in both a fundamental understanding of such applications and to push forward

the development and uses of ultrasound techniques for cement-based composites. Amongst these the authors suggest the following lines of investigation:

- The effect of PUS on Portland cement hydration is not yet fully understood. More studies need to be conducted to confirm the effects of cavitation on cementitious materials, and at the same time clarify its mechanisms. The only undertaken study has focused on limited characterisation of alite hydration which is only considered as a binary system of Ca and Si ions in cement hydration. However, the effect of exposing PUS to complex systems of ternary compositions of CaO-SiO_2-Al_2O_3 need to be thoroughly investigated. Overall, the authors believe that it is now time to expand the horizon of ultrasound-assisted crystallisation studies known as "sonocrystallisation" to cement hydration products. The aim should be to develop advanced theories regarding the homogenous and heterogeneous nucleation and growth of cement hydration affected by ultrasound irradiation.
- De-agglomeration and gaining an enhanced particle size distribution has been the primary focus of nearly all studies carried out in the use of PUS to promote SCMs properties. In addition, microstructural studies on cementitious composites have failed to reveal an in-depth analysis of the hydration as well as mechanical and durability properties characterisation. Therefore, it is necessary to study the kinetics and mechanism of PUS affected cementitious blended systems in which the hydraulic reaction of SCMs could lead to more complex hydration phases in combination with cavitational effects. The first step in fulfilling this objective may be to develop an analytical approach to qualify and quantify the reaction of SCMs independently of the cement clinker phases. Then, the intermixing levels of hydration phase compositions in blended systems (i.e. PC-silica fume, PC-slag, PC-PFA, PC-natural pozzolan) subjected to PUS should be investigated.
- The fresh state properties of cementitious materials markedly dominate the practical aspects of their usage in the field. In addition to the initial setting time, the characterisation of rheological parameters (i.e. yield stress and viscosity) of sonicated cement-based systems need to be well established.
- Expanding the practice of sonicated cement paste to concrete system should be pursued in future studies. It would require the development and manufacturing of ultrasound equipment tailored for concrete, and more notably, scaled up sonochemical processes.
- The influence of the degassing (de-airing or de-foaming) effect of PUS especially in present of air-entrained admixtures needs to be investigated.
- Studies on the influence of PUS on the crystallisation of hydration products in the presence of superplasticisers needs to be performed.
- The study regarding the influence of high frequency as well as combined low and high frequency ultrasound exposure on sonocrystallisation of cementitious materials hydration needs to be carried out.
- The generated heat due to the cavitation phenomenon during the PUS practice which could adversely affect initial setting needs to be addressed.

It is hoped that further research progress in this field will drive the commercial utilisation of PUS in the construction industry.

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